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A Versatile High Pressure Test Bench for Supercritical Fluid Research

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Research involving critical fluids is a sub-discipline of high pressure technology, and should also encompass the evaluation of components (pressure gauges, flowmeters, pumps, etc.) that are routinely used in measuring experimental data and mechanically conducting extractions or reactions. A unique system has been developed at NCAUR for performing these tasks, which can include the calibration of various types of pressure gauges, determining the leak status of high pressure valves, or testing the efficacy of repaired pumps or compressors used for fluid delivery. The pressure test bench is capable of generating reproducible pressures to 20,000 psi, and features both low and high calibration gauges which are routinely used in the NCAUR High Pressure Laboratory. The bench is capable of both producing and/or receiving pressure signals, allowing it to be used for many types of pressure calibration and testing for fluid leakage. A description of the design, construction, and use of this pressure test bench will be presented, including provision for automatic data recording of pressure, flow, and other variables which impact on using critical fluids in a high pressure laboratory.

Introduction

Various high pressure components, such as pressure gauges, transducers, relief valves, shut-off valves, micrometering valves, and gas booster pumps, are used at the High Pressure Laboratory of NCAUR to construct small scale pilot plant apparatus. References pertaining to the control systems and a variety of apparatus constructed at the High Pressure Laboratory can be found in previously published literature.[1,2] The High Pressure Laboratory is well stocked with new as well as used components. The unknown condition of the many used components and the desire to check the performance of repaired pumps precipitated the need for a pressure test bench. It should be noted that it was not our intent to design or construct a test bench to proof test or burst test pressure vessels. Photographs of the completed test bench, front and back, are shown in Figures 1 and 2.

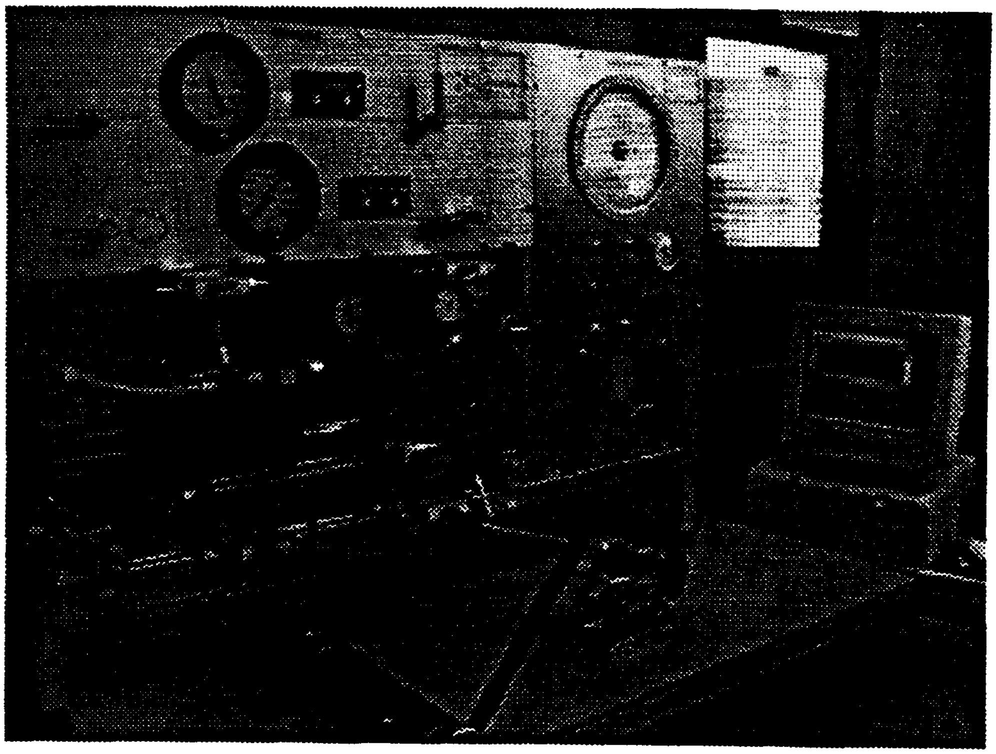

Figure 1. Photograph of front view of pressure test bench.

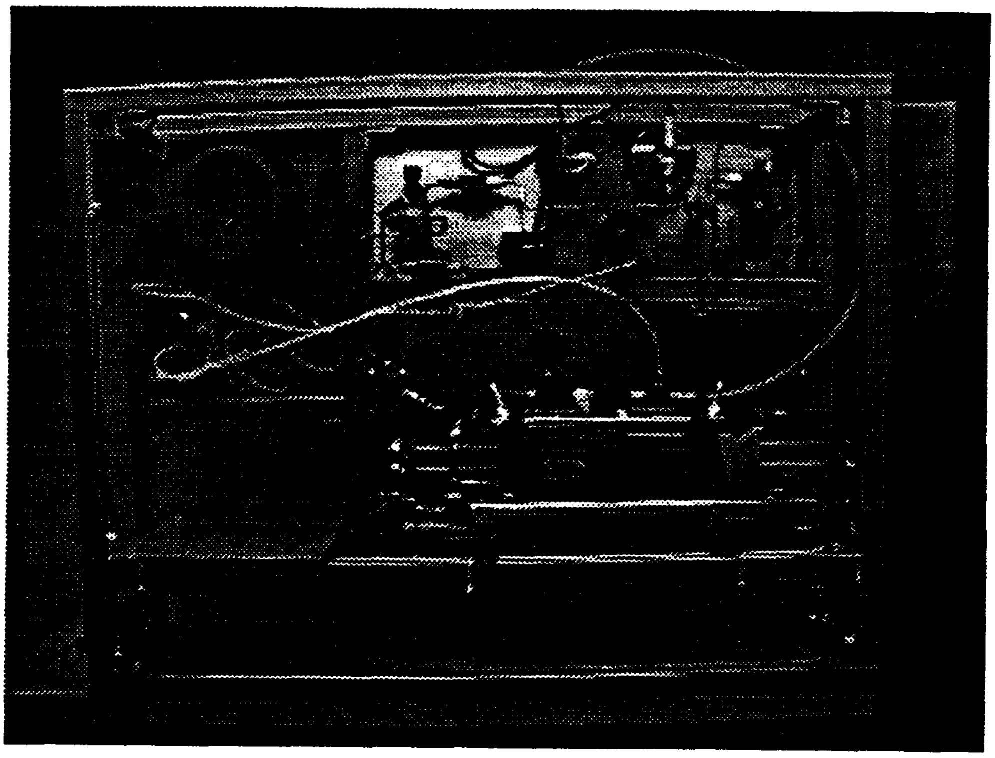

Figure 2. Photograph of back view of pressure test bench.

Design

The design for the pressure test bench was finalized after giving consideration to safety, functionality, and available existing components. A schematic of the test bench is shown in Figure 3.

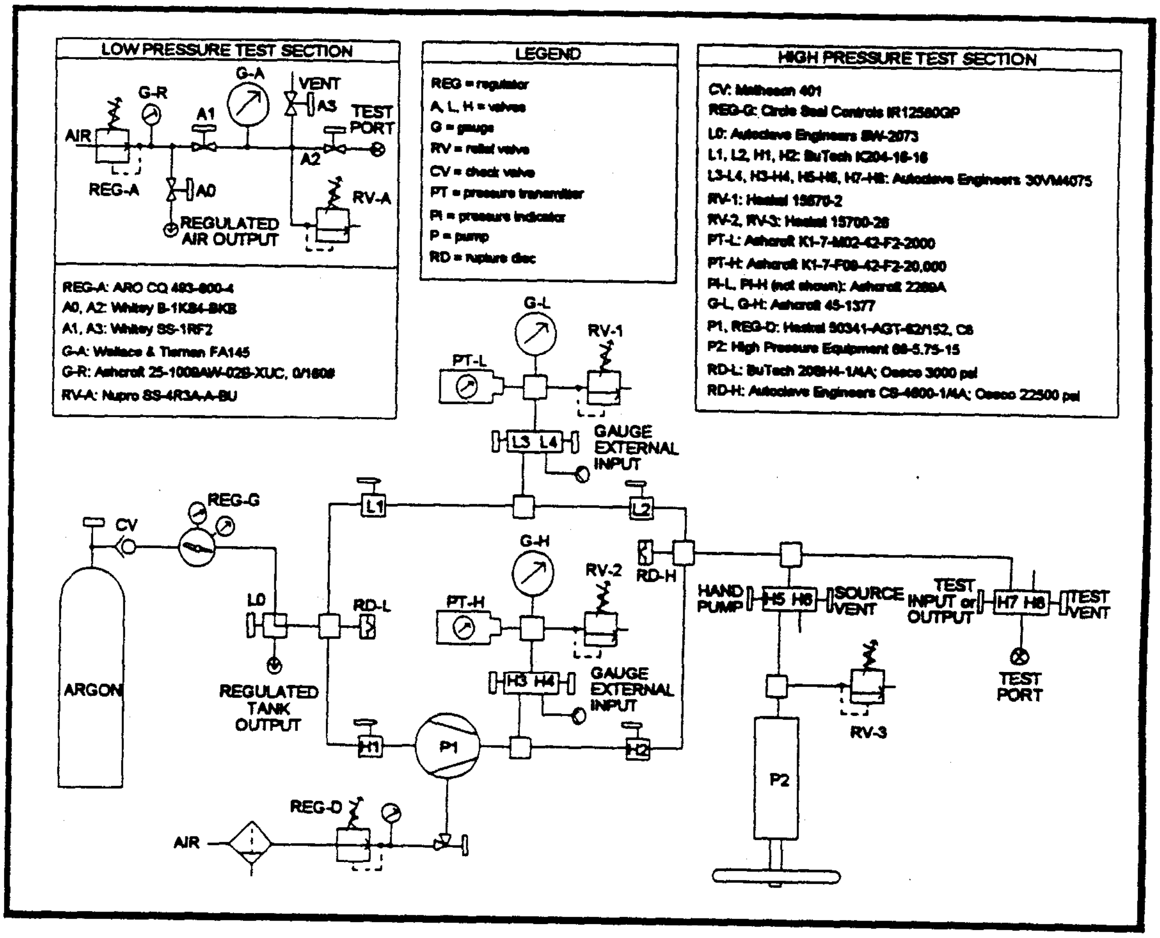

Figure 3. Schematic of the pressure test bench.

The test bench was divided into two sections: one for low pressure testing (using compressed air) and one for high pressure testing (using a cylinder tank of argon). The low pressure test section provides controlled compressed air up to 150 psi, supplied by the High Pressure Laboratory's dedicated air compressor (2T25A-240; Emglo, Johnstown, PA). The high pressure test section has the capability to provide cylinder tank pressure through a precision regulator up to 2000 psi. Higher pressures up to 20,000 psi are obtained through the use of an air-driven gas booster pump. Precision adjustments of pressures up to 15,000 psi are allowed on the high pressure test section through the use of a hand pump pressure generator. In consideration of safety and to protect components of the test bench, relief valves and rupture discs were included in the design. An electrical signals connection panel was also included to allow for data acquisition of the pressure signals and calibration of the high pressure test section's digital pressure indicators. The performance of various components, such as those that accept pressure and those that generate pressure, can be checked at the test bench. Design is such that leak checking of components at pressures up to 15,000 psi can be accomplished by observance of a pressure drop (bleed rate).

Construction

Certain components were chosen for inclusion in the test bench, such as the gas booster pump and the hand pump pressure generator, to duplicate components already used in the High Pressure Laboratory. Many of the remaining components were selected based on the High Pressure Laboratory's stock of existing parts. If purchased new, the total cost for components and parts used in the construction of this test bench would be approximately $15,800. Tables 1 and 2 provide information on all of the components incorporated in the test bench.

Table 1. Pressure test bench components.

ITEM [DRAWING REFERENCE]	MANUFACTURER	PART NUMBER	PRICE	QTY.	TOTAL
air regulator [REG-A]	ARO	CQ493-600-4	$34.59	1	$34.59
needle valve [A0, A2]	Whitey	B-1KS4-BKB	30.80	2	61.20
needle valve [A1, A3]	Whitey	SS-1RF2	46.40	2	92.80
pressure gauge [G-A]	Wallace & Tiernan	FA145 (62A-2A-0100)	1525.00	1	1525.00
pressure gauge [G-R]	Ashcroft	25-1009AW-02B-XUC, 0/160#	55.70	1	55.70
relief valve [RV-A]	Nupro	SS-4R3A-A-BU	124.15	1	124.15
1/4-inch tube to 1/4-inch NPT male elbow	Swagelok	B-400-2-4	3.60	3	10.80
1/4-inch tube to 1/4-inch NPT female elbow	Swagelok	B-400-8-4	4.80	1	4.80
1/4-inch tube union tee	Swagelok	B-400-3	5.70	4	22.80
1/4-inch tube bulkhead union	Swagelok	B-400-61	3.50	3	10.50
1/4-inch tube to 1/8-inch NPT male connector	Swagelok	SS-400-1-2	4.70	4	18.80
1/2-inch NPT to 1/4-inch NPT bushing			0.95	2	1.90
1/4-inch plastic tubing (per foot)	Weatherhead	PT-240-04	0.10	14	1.40
gas cylinder support clamp	Fisher Scientific	10-595	29.54	1	29.54
argon, 220 cubic feet industrial grade	BOC Gases		18.48	1	18.48
check valve [CV]	Matheson	401V	93.00	1	93.00
tank regulator [REG-G]	Circle Seal	IR12580GP	526.00	1	526.00
2-way straight valve [L0]	Autoclave Engineers	SW-2073	105.00	1	105.00
ball valve [L1, L2, H1, H2]	BuTech	K204-16-16	180.00	4	720.00
3-way/2-stem manifold valve [L3-L4, H3-H4, H5-H6, H7-H8]	Autoclave Engineers	30VM4075	140.00	4	560.00
relief valve [RV-1]	Haskel	15670-2	195.00	1	195.00
relief valve [RV-2, RV-3]	Haskel	15700-26	561.00	2	1122.00
pressure transmitter [PT-L]	Ashcroft	K1-7-M02-42-F2-2000	213.30	1	213.30
pressure transmitter [PT-H]	Ashcroft	K1-7-F09-42-F2-20,000	253.80	1	253.80
digital panel meter [PI-L, PI-H]	Ashcroft	2289A	280.25	2	560.50
pressure gauge [G-L]	Ashcroft	45-1377SS-02B, 0/2000#	205.70	1	205.70
pressure gauge [G-H]	Ashcroft	45-1377SS-09B, 0/20,000#	363.10	1	363.10
gas booster pump [P1, REG-D]	Haskel	50341-AGT-62/152, C8	5123.00	1	5123.00
pressure generator [P2]	High Pressure Equipment	68-5.75-15	1950.00	1	1950.00
safety head [RD-L]	BuTech	208H4-1/4A	71.00	1	71.00
rupture disk [RD-L]	Oseco	S-1843-01	21.80	1	21.80
safety head [RD-H]	Autoclave Engineers	CS-4600-1/4A	66.00	1	66.00
rupture disk [RD-H]	Oseco	R-6884-01	32.30	1	32.30
1/8-inch tube to 1/4-inch NPT male connector	Swagelok	SS-200-1-4	5.80	2	11.60
1/8-inch tube bulkhead union	Swagelok	SS-200-61	15.80	1	15.80
1/4-inch MP tee	Autoclave Engineers	CTX4440	42.00	2	84.00
1/4-inch MP cross	Autoclave Engineers	CXX4444	48.00	1	48.00
1/4-inch HP tee	Autoclave Engineers	CT4440	52.00	4	208.00
1/4-inch HP cross	Autoclave Engineers	CX4444	58.00	2	116.00
1/4-inch HP plug	Autoclave Engineers	AP40	4.70	1	4.70
M/F adapter 1/4-inch NPT to 1/4-inch HP	Autoclave Engineers	6M44N3	23.50	1	23.50
M/F adapter 1/8-inch LP to 1/4-inch HP	Autoclave Engineers	6M24C3	23.00	2	46.00
M/F adapter 1/4-inch NPT to 1/4-inch MP	Autoclave Engineers	6M44N6	23.50	2	47.00
1/4-inch NPT cross	Cajon	SS-4-CS	29.40	1	29.40
1/4-inch NPT plug			1.53	2	3.06
1/4-inch MP tube (per foot)	Autoclave Engineers	MS15-092	8.50	7.3	62.05
1/4-inch HP tube (per foot)	Autoclave Engineers	MS15-081	9.00	33.7	303.30
1/8-inch SS tubing	Restek	21512	1.86	5	9.30
1/2-inch NPT to 3/8-inch NPT bushing			0.73	1	0.73
3/8-inch tube to 3/8-inch NPT male connector	Swagelok	B-600-1-6	2.40	1	2.40
cabinet style work bench with laminated hardwood top	Lyon Metal Products	2844 (60x28x34 inches)	327.63	1	327.63
48x48x1/8-inch alloy 5052 aluminum sheet	McMaster-Carr	88895K76	88.00	1	88.00
1 5/8x1 5/8-inchx12-gauge metal framing channel (per foot)	B-Line	B22	1.34	46	61.41
1/4x1 1/4-inch lag screw			0.30	7	2.10
1/4-inch flat washer			0.01	11	0.16
two-hole corner angle	B-Line	B101	0.92	13	11.96
two-hole "no-twist" corner angle	B-Line	B231	1.20	5	6.00
square washer	B-Line	B200	0.54	5	2.70
three hole gusseted shelf angle	B-Line	B125	2.95	1	2.95
four hole corner plate	B-Line	B143	3.35	4	13.40
1/4-20 channel nut	B-Line	TN224	0.98	8	7.84
1/4-20x3/4-inch hex head cap screw			0.04	34	1.22
1/2-13 channel nut	B-Line	N225	0.71	27	19.17
1/2-13x1 1/4-inch hex head cap screw	B-Line	HHCS 1/2" x 1 1/4"	0.25	46	11.50
1 1/4-inch pipe clamp	B-Line	B2211	0.89	1	0.89
1 1/2-inch pipe clamp	B-Line	B2212	0.92	2	1.84
1/4-20x1/2-inch round head slotted screw			0.23	8	1.86
3/8-16 channel nut	B-Line	N228	0.61	4	2.42
3/8-16x1-inch hex head cap screw			0.09	4	0.37
8-32x5/8-inch oval head Phillips screw			0.04	3	0.13
10-24x1 1/4-inch socket head cap screw			0.30	8	2.40
M4x10mm socket head cap screw			0.25	2	0.50
3/8-16x3-inch hex head cap screw			0.22	2	0.43
3/8-inch flat washer			0.02	8	0.16
3/8-inch split-lock washer			0.02	2	0.03
3/8-16 machine screw nut			0.05	2	0.09
double banana plug	Pomona	1330	2.25	4	9.00
panel mount banana jack	Pomona	1581	0.85	8	6.80
4-conductor shielded cable (per foot)	General Cable	C0762	0.27	10	2.66
6-foot 18/2 AC line cord			1.61	2	3.22
4x4x2 1/8-inch outlet box			1.08	1	1.08
switch/duplex box cover			0.79	1	0.79
switch			0.45	1	0.45
duplex receptacle			0.35	1	0.35
10-foot 14/3 AC line cord			7.56	1	7.56
TOTAL					$15,807.29

Table 2. Sources for components used in construction of the pressure test bench.

COMPANY [PARENT COMPANY]	LOCATION	TELEPHONE	INTERNET ADDRESS
ARO [Ingersoll-Rand]	Bryan, OH	(419) 636-4242	http://www.aro.ingersoll-rand.com/
Ashcroft [Dresser Instrument Division]	Stratford, CT	(203) 378-8281	http://www.dresser.com/instruments/
Autoclave Engineers [Snap-tite]	Erie, PA	(814)-838-5700	http://www.snap-tite.com/ae/
B-Line [Sigma-Aldrich]	Highland, IL	(618) 654-2184	http://www.bline.com/
BOC Gases	Murray Hill, NJ	(908) 464 8100	http://www.bocgases.com/
BuTech Pressure Systems	Erie, PA	(814) 833-4904	http://www.ncinter.net/~butech/
Cajon [Swagelok]	Macedonia, OH	(440) 349-5934	http://www.swagelok.com/
Circle Seal Controls	Corona, CA	(909) 270-6200	http://www.circle-seal.com/
Fisher Scientific	Pittsburgh, PA	(412) 490-8300	http://www.fishersci.com/
General Cable	Highland Heights, KY	(606) 572-8000	http://www.generalcable.com/
Haskel International	Burbank, CA	(818) 843-4000	http://www.haskel.com/
High Pressure Equipment	Erie, PA	(814) 838-2028	http://www.highpressure.com/
Lyon Metal Products	Aurora, IL	(630) 892-8941	http://www.lyonmetal.com/
Matheson Tri-Gas	Parsippany, NJ	(973) 257-1100	http://www.mathesongas.com/
McMaster-Carr	Elmhurst, IL	(630) 833-0300	http://www.mcmaster.com/
Nupro [Swagelok]	Willoughby, OH	(440) 349-5934	http://www.swagelok.com/
Oseco	Broken Arrow, OK	(918) 258-5626	
Pomona	Pomona, CA	(909) 469-2900	http://www.pomonaelectronics.com/
Restek	Bellefonte, PA	(814) 353-1300	http://www.restekcorp.com/
Swagelok	Solon, OH	(440) 349-5934	http://www.swagelok.com/
Wallace & Tiernan	Belleville, NJ	(973) 759-8000	http://www.wandt.com/
Weatherhead [Dana]	Toledo, OH	(419) 891-2800	http://www.dana.com/weatherhead/
Whitey [Swagelok]	Highland Heights, OH	(440) 349-5934	http://www.swagelok.com/

The versatility of channel strut metal framing was used to an advantage for constructing the framework of the test bench. Components were mounted on the open framework built with 1-5/8 inch square galvanized steel channel strut (B22; B-Line). This frame was attached to a 60-inch by 28-inch wood-top workbench. Dimensions for the frame were determined after taking into consideration the space needed for the various components. Aluminum panels, 1/8-inch thick and sheared to size, were attached to the frame to accommodate panel-mount gauges and valves. A 14-inch by 12-inch aluminum panel was also sheared and installed as a shelf below the low pressure test section.

Dimensional layout of the frame and panels was performed using a Computer Aided Design (CAD) software program, "TurboCAD for Windows" version 1.02 (IMSI, San Rafael, CA). Outlines of the various components were drawn at 4:1 or 6:1 scales and arranged to create a functional and aesthetically pleasing layout. These drawings are shown in Figures 4, 5, 6, and 7.

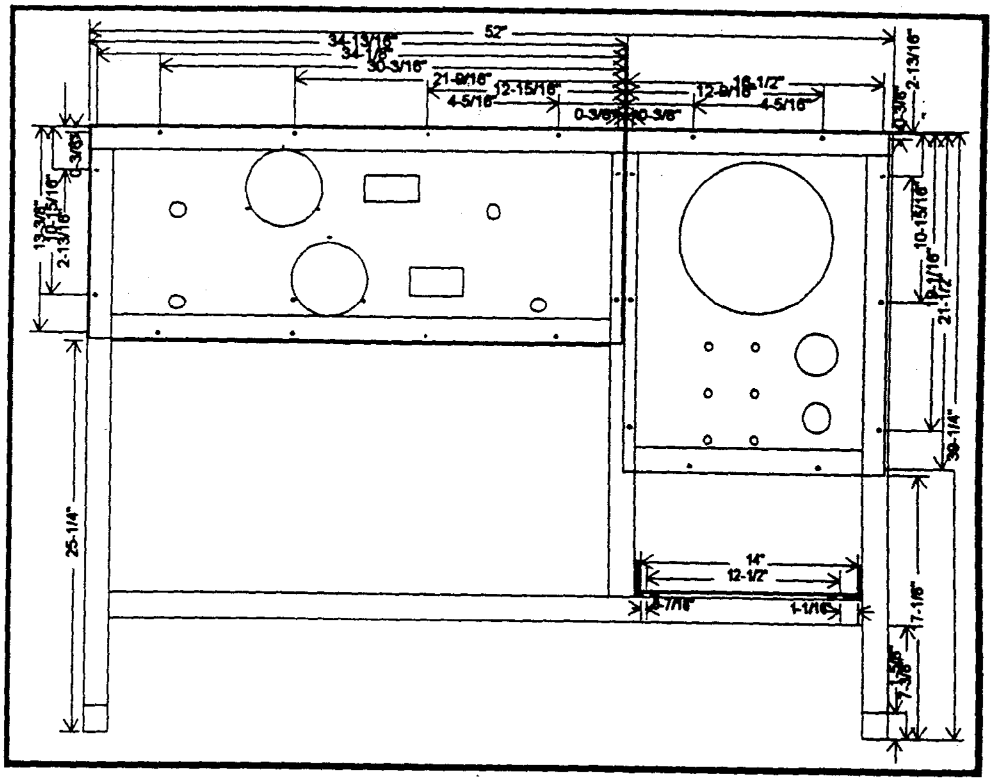

Figure 4. Frame layout.

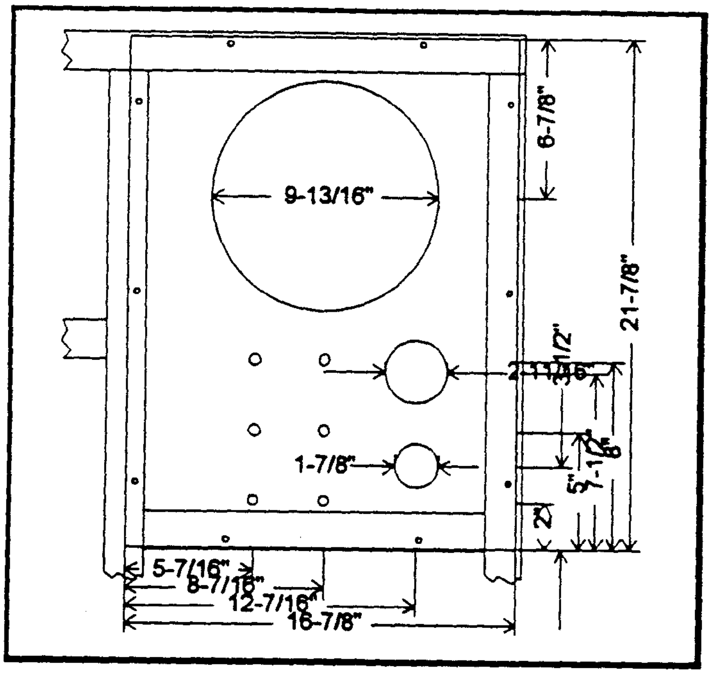

Figure 5. Low pressure panel layout.

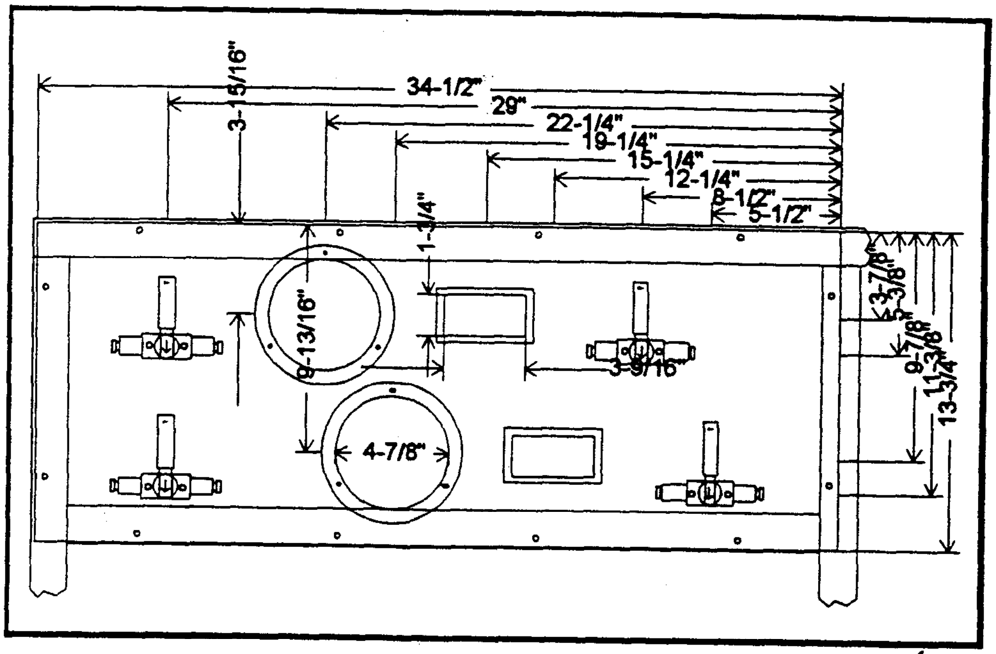

Figure 6. High pressure panel layout.

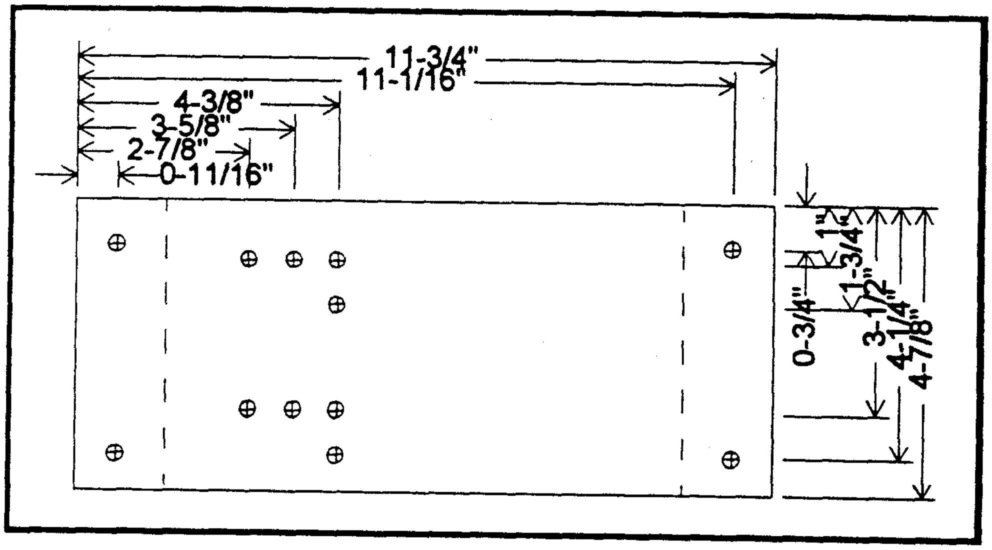

Figure 7. Electrical signals connection panel layout.

Dimensions taken directly from the CAD drawings were used to fabricate the channel strut frame and aluminum panels. The framework outside dimensions are 52 inches wide, 12 inches deep, and 39-1/4 inches high. Corner angle channel strut components (B231, B101; B-Line) were used to fasten the lengths of channel strut together to make the frame.

The low pressure test section incorporates a surplus precision 100 psi, 8-1/2 inch diameter, double-revolution dial pressure gauge [G-A]. A relief valve [RV-A], set at 100 psi, protects this gauge from over pressurization. One-quarter inch bulkhead unions (B-400-61; Swagelok) provide convenient test port connection points. One-quarter inch plastic tubing (PT-240-04; Weatherhead) was used to connect components behind this panel.

Four existing ball valves [L1, L2, H1, H2] were mounted on the panel of the high pressure test section, along with analog and digital pressure gauges. Existing 3-way/2-stem manifold valves [L3-L4, H3-H4, H5-H6, H7-H8] were used to advantage, since they reduced the number of tube connections. These valves were mounted on four-hole corner plates (B143; B-Line) modified with two 10-24 tapped holes. This provided a secure mounting for the valves through a simple connection to the frame using 1/2-13 hex head cap screws (HHCS 1/2" x 1 1/4"; B-Line) and channel nuts (N225; B-Line). Relief valves [RV-1, RV-2, RV-3] to protect the gauges [G-L, G-H], the transmitters [PT-L, PT-H], and the pressure generator [P2] were mounted to the frame with channel strut pipe clamps (B2211, B2212; B-Line). A 1-1/2 inch diameter hole was cut into a three-hole gussetted shelf angle (B125; B-Line) to accommodate mounting of the tank regulator [REG-G]. One-quarter inch O.D., 0.083 inch I.D. high pressure tube, rated for 60,000 psi (MS15-081; Autoclave Engineers), and 1/4 inch O.D., 0.109 inch I.D. medium pressure tube, rated for 20,000 psi (MS15-092; Autoclave Engineers), were used to interconnect components in this section. The one exception was from the cylinder tank check valve [CV] to the input side of the tank regulator [REG-G], where 1/8 inch O.D., 0.085 inch I.D. stainless steel tubing (21512; Restek) was used.

Performance

External pressure connections to the low pressure test section are made through 1/4 inch compression fittings (Swagelok -400 connection type). Adapters have been made to accommodate connections to components with other type connection ports.

External pressure connections to the high pressure test section are made through 1/4 inch standard high pressure cone and thread connections (Autoclave Engineers F250C connection type). The one exception is at the regulated tank output valve [L0], where the output connection is made with a 1/8 inch gland and compression sleeve (Autoclave Engineers W125 connection type). Connection adapters have also been made for this test section.

The pressure test bench permits a multitude of test configurations. Graphic layouts, shown for the high pressure test panel in Figure 8, were drawn on both panels with permanent markers to indicate device interconnections for straightforward use of the test bench.

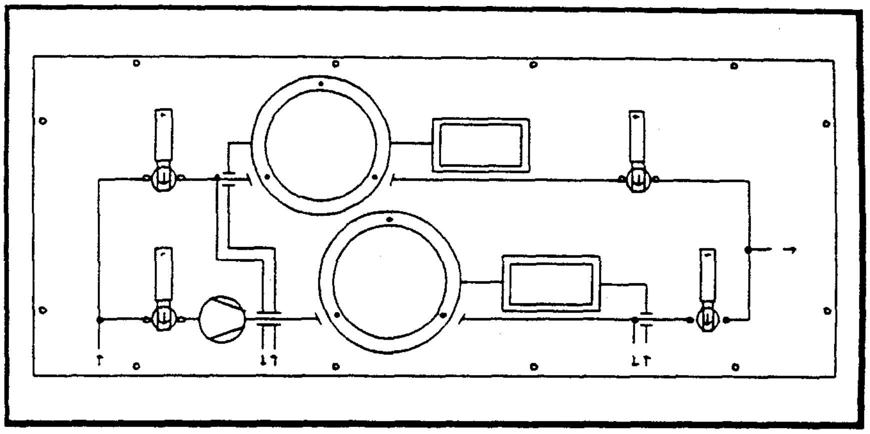

Figure 8. High pressure test panel graphic layout.

Though operation of the test bench is intuitive, step-by-step test procedures have been developed for various components. Figure 9 shows one of these procedures.

Procedure using low and high pressure test sections to test an air-driven gas booster pump:
· Make sure air regulator REG-A and tank regulator REG-G are adjusted fully counterclockwise.
· Connect gas input of pump-under-test to REGULATED TANK OUTPUT port.
· Connect gas output of pump-under-test to 20,000 psi GAUGE EXTERNAL INPUT port.
· Connect air drive input of pump-under-test to REGULATED AIR OUTPUT port.
· Close valves A1, L2, L4, H1, H5, H6, H7.
· Open valves A0, L0, L1, L3, H2, H3, H4.
· Adjust tank regulator REG-G for desired gas input pressure for pump-under-test as observed on gauge G-L and indicator PI-L.
· Adjust air regulator REG-A to obtain desired gas output pressure for pump-under-test as observed on gauge G-H and indicator PI-H.
· Open valve H6 when it is desired to vent gas output pressure for pump-under-test.
· Before disconnecting pump-under-test, adjust air regulator REG-A and tank regulator REG-G fully counterclockwise and open valve H6 .

Figure 9. Sample test procedure.

One consideration needs to be noted when using the gas booster pump [P1] and the hand pump pressure generator [P2]. Adjustments made with the pressure generator [P2] to lower the test pressure below the set pressure of the booster pump [P1] will be canceled out by the compensating action of the booster pump [P1] returning to its set pressure. Therefore, the pressure setting of the booster pump [P1] needs to be kept lower than the pressure that the pressure generator [P2] will be adjusted to. (Increasing test pressures generated by the pressure generator [P2] are not compensated by the booster pump [P1] due to operation of the booster pump's [P1] internal check valve.)

As with any system that develops high pressures, intended actions need to be thought through with safety in mind. Venting of test gases away from personnel is a must. All vent valves on this test bench discharge to the back of the bench, away from personnel. Tubing used to connect the test bench to the device-under-test must be adequately rated for the pressure that will be applied. Connections should not be broken until all pressure has been relieved from the device-under-test. Testing should always start with all pressure regulator knobs turned fully counterclockwise (zero pressure). The appropriate responsive action to an unexpected failure of a device-under-test should be determined before a test procedure is initiated.

Data Acquisition

Provisions were made through the electrical signals connection panel to measure and record the 4 to 20 mA signals from the pressure transmitters [PT-L, PT-H]. A schematic showing the wiring and operation of one of the two identical channels of the electrical signals connection panel is shown in Figure 10.

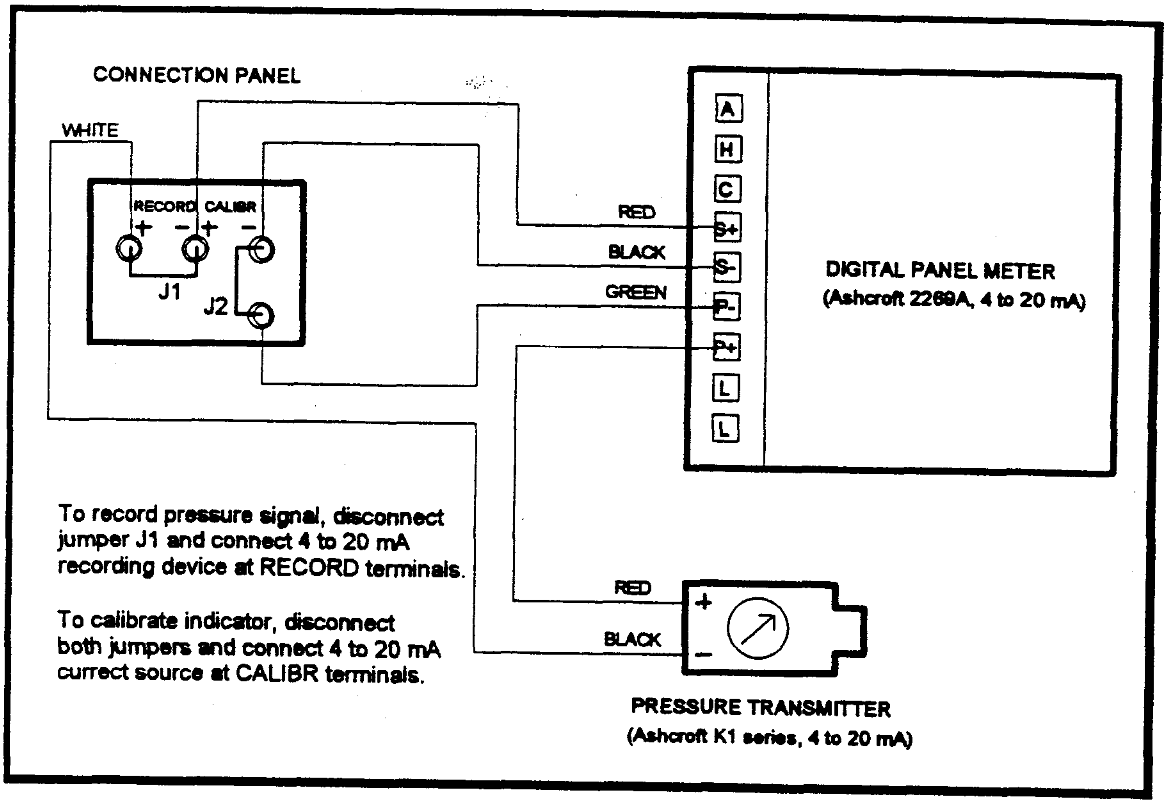

Figure 10. Schematic for one-half of the electrical signals connection panel.

Data was recorded during a test of a used relief valve that provides an example of this test bench feature. This data was collected from the 20,000 psi pressure transmitter [PT-H] using National Instruments (Austin, TX) SCXI hardware and LabVIEW version 5.0.1 software. Figure 11 shows the pressure relieving performance of the used relief valve set at approximately 9000 psi and checked in two different ways.

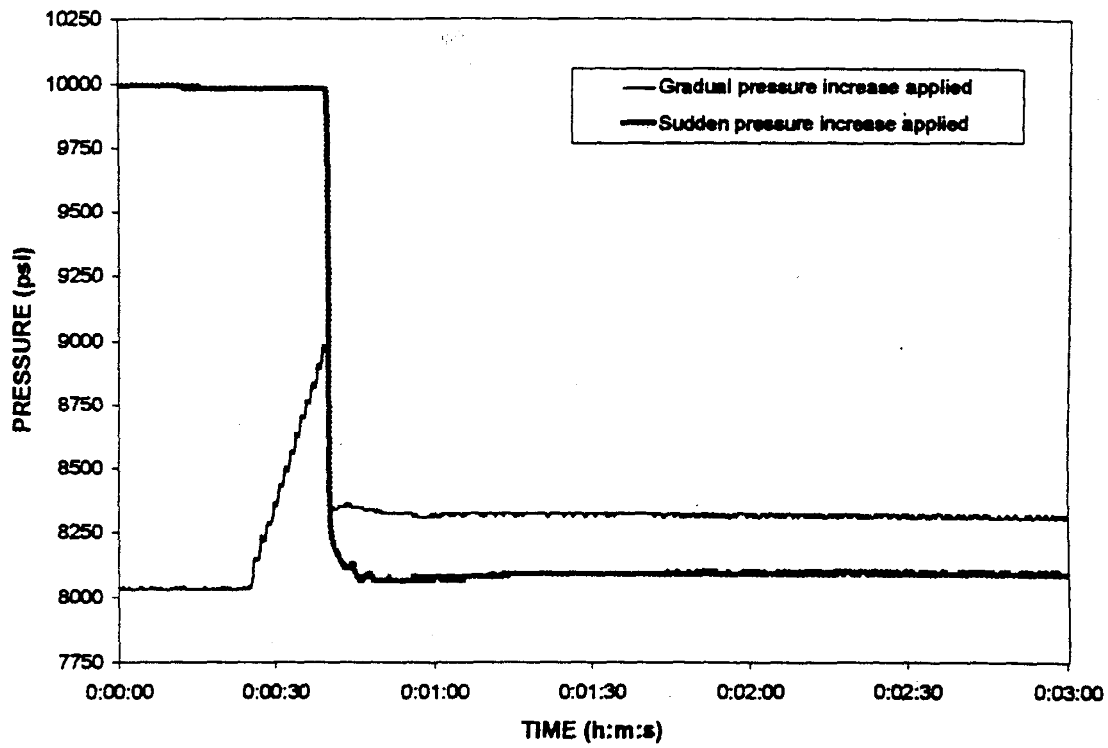

Figure 11. Performance of relief valve-under-test.

The first method applied a gradual increasing pressure to the relief valve: Booster pump [P1] was used to pressurize the relief valve, connected at the TEST PORT of test output valve [H7], to an initial 8000 psi. Then pressure generator [P2] was rotated clockwise (approximately 14 half-turns) to actuate the relief valve. The second method applied a sudden over-pressurization of 10,000 psi to the relief valve: Booster pump [P1] was used to pressurize the relief valve to 8000 psi. Next, test output valve [H7] was closed, and the test bench was pressurized to 10,000 psi. After turning drive regulator [REG-D] fully counterclockwise, test output valve [H7] was quickly opened, permitting actuation of the relief valve.

The electrical signal connections panel also provides a convenient manner for external calibration of the digital panel meters (indicators) [PI-L, PI-H] with a current-sourcing 4 to 20 mA calibrator.

Conclusion

Components utilized in supercritical fluid research are often incorporated without initially testing their condition. Used components, as described previously, should be routinely tested to see if they are in a safe condition and meet operational specifications. The described pressure test bench was built by taking advantage of existing parts and incorporating components that are used in the NCAUR Critical Fluids Research Program. The pressure test bench is designed specifically for evaluating components routinely used in supercritical fluid research.

In the future a mass flowmeter will be added to the test bench to provide flow calibration capability. Performance of the described test bench has met desired expectations and supplied the laboratory with a needed testing apparatus.

Acknowledgments

Names are necessary to report factually on available data; however, the USDA neither guarantees nor warrants the standard of the product, and the use of the name by USDA implies no approval of the product to the exclusion of others that may also be suitable.

(1) Friedrich, J. P. Wheels Make High-Pressure Research Roll. *Annals of the New York Academy of Sciences* **1970**, *172*, 155-174.

(2) King, J. W. Analytical-Process Supercritical Fluid Extraction: A Synergistic Combination for Solving Analytical and Laboratory Scale Problems. *Trends in Anal. Chem.* **1995**, *14*, 474-481.

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